

EFINS-66-47

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THE SURVEYOR LUNAR MISSION

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GPO PRICE \$
CFSTI PRICE(S) \$
Hard copy (HC) 2.50
Microfiche (MF) .50

FF 653 July 65

N 67 13243	
(ACCESSION NUMBER)	(THRU)
30	1
(PAGES)	(CODE)
CR 75748	30
(NASA CR OR TMX OR AD NUMBER)	(CATEGORY)

Submitted to
The Journal of Geophysical Research
May 1966

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ABSTRACT

An experiment has been designed to determine the chemical composition of the lunar surface on the Scientific Surveyor soft-landing missions to the moon. The instrument employed utilizes the characteristic spectra of backscattered alpha particles and protons from (α ,p) reactions to establish the elements present in a sample. The instrument can determine the amounts of most elements present in rocks with a sensitivity and accuracy of about one atomic per cent. Satisfactory analyses have been obtained of a variety of terrestrial samples.

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INTRODUCTION

The chemical composition of an extraterrestrial body (for example, the moon) is one of the most important scientific facts to be learned about such a body. A chemical analysis would provide a clue to the history and present stage of development of the moon; it would probably settle the question whether any of the meteorites falling on the earth have a lunar origin. On the technological side, a chemical analysis would give the first experimental information on the raw materials available. It might also indicate the chemical reactivity of the surface materials.

In the early stages of lunar investigation, the chemical analysis that is desired is one which is general enough to detect unusual and unexpected chemical compositions, and yet precise enough to distinguish between compositions similar to those of the gross classes of terrestrial rocks. In order to achieve this objective on an unmanned, instrumented mission, such as the Surveyor series, with its severe limitations on weight, power, and complexity of operations, a new method of chemical analysis has been developed [Turkevich, 1961; Patterson, Turkevich, and Franzgrote, 1965] and instrumented [Turkevich, Knolle, Emmert, Anderson, Patterson, and Franzgrote, 1966]. In this report, we give only a summary of the principles of the analytical method and concentrate on the adaptation of the instrument to the Surveyor mission requirements.

This new technique of analysis takes advantage of the characteristic interactions of alpha particles with matter to provide information on the chemical composition. The energy spectra of the large-angle, elastically-scattered alpha particles are characteristic of the nuclei doing the scattering. In addition, certain elements, when bombarded with alpha particles, produce protons, again with characteristic energy spectra. Consequently, these energy spectra and intensities of scattered alpha particles and protons can be used to determine the chemical composition of the material being exposed to the alpha particles.

The method has good resolution for the light elements expected in rocks (unfortunately, however, it can give only indirect information about hydrogen). The resolution becomes poorer as the atomic weight increases (Fe, Co, and Ni cannot easily be resolved), even though the sensitivity is greater for heavy than for most light elements. The approximate sensitivity for elements heavier than boron is about one atomic per cent.

The absence of an atmosphere on the moon makes practical the use, for such chemical analyses, of the relatively low-energy alpha particles from a radioactive source.

Cm^{242} ($t_{1/2} = 163\text{d}$, $T_{\alpha} = 6.11\text{ MeV}$) is a convenient nuclide for this purpose. However, the use of low-energy alpha particles restricts the information obtained to that pertaining to the uppermost few microns of material. The method is thus one of surface chemical analysis. Moreover, using practical source

intensities (~ 100 mc.), the rate of analysis is rather slow: a relatively complete one requires about one day. In spite of these disadvantages, the simplicity of the instrumentation associated with using a radioactive source makes this an attractive approach for the first Scientific Surveyor Missions.

DESCRIPTION OF THE INSTRUMENT

An instrument based on these principles and designed for space missions is described elsewhere [Turkevich, Knolle, Emmert, Anderson, Patterson, and Franzgrote, 1966]. The version presently planned for Surveyor is identical in design as regards the basic physical phenomena. It differs primarily in the packaging, which has been modified to ensure better passive temperature control [Walker and Wolf, 1966]. In addition, an on-site electronic calibration mode has been incorporated.

The instrument consists of two principal units: a deployable "head" and an electronics package. Figure 1 shows the Type Approval Models of these units. The head is primarily a box ($6\frac{3}{4}$ " x $6\frac{1}{2}$ " x $5\frac{1}{4}$ " high), with a 12" diameter plate on the bottom. The electronics unit is a box (7" x $6\frac{1}{2}$ " x 4" high). The bare weights (without cabling or harness) are 4.7 and 4.8 lbs., respectively. The instrument uses less than 1.2 watts in operation.

The head of the instrument is to sit on the spacecraft until after lunar landing. Then, after certain preliminary measurements, it is to be deployed onto the surface. The purpose of the 12" plate at the bottom is to minimize the probability of the box sinking appreciably into a possibly soft lunar surface. In the bottom of the head is a 4-1/4" circular opening. Recessed 2-3/4" above the opening are a set of six alpha sources, which are packaged in such a way that the alpha particles strike only the opening of the head. Close to the alpha sources are two silicon semiconductor detectors arranged to detect alpha particles scattered back from the opening at an average angle of 174° . These two detectors, together with their associated electronics, can register both the number and the energy of the alpha particles scattered back to them.

In the head, also, are four detectors ($\sim 1 \text{ cm}^2$ area each) designed to detect protons produced in the sample by the alpha particles. A gold foil of $\sim 21 \text{ mg cm}^{-2}$ prevents scattered alpha particles from reaching these detectors. Together with the associated electronics, these detectors determine the number and energy of protons produced in the sample. Because the expected proton rates are low, and because these detectors are particularly sensitive to solar protons, the proton detectors are backed by other detectors. The electronics associated with these guard detectors are arranged so that an event registered in both (and, therefore, due to space radiation) will not be considered as coming from the sample. This arrangement is expected to reduce significantly the backgrounds in the proton mode of the instrument.

In addition to the sources, detectors, and associated electronics, the head contains a temperature sensor, a 5-watt heater, and an electronically-actuated mechanical pulser. This latter can be used to calibrate the electronics of the instrument by introducing electrical pulses of two known magnitudes at the detector stages of the systems. This calibration mode can be initiated by command from earth.

The output of the head is thus a signal (in time analogue form), which characterizes the energy of an event in either the scattered alpha or proton mode of the instrument. The electronics unit processes this signal, converting it to digital form and, after two stages of data smoothing, makes it available for spacecraft transmission to earth. The energy spectra are expressed in terms of a 128 channel pulse-height analyzer having a threshold of about 600 KeV and a gain of about 54 KeV per channel.

At a total source strength of 10^{11} d/m, the data rates expected, from rocks of typical composition, are about one event per sec in the alpha mode and about a tenth of this in the proton mode. The characteristics of each event are to be transmitted, with essentially no spacecraft storage, down to earth at a rate of $2200 \text{ bits sec}^{-1}$ for the alpha mode and $550 \text{ bits sec}^{-1}$ for the proton mode.

The electronics package has, in addition to the digital processing electronics, power supplies and the logical interfaces between the instrument and the spacecraft. For example, the outputs of the individual detectors, together with their associated guard detectors, can be blocked by command from earth. Via the electronics unit, also, the temperature of the head, as well as various monitoring voltages, can be transmitted down to earth. Finally, a crude rate-meter provides information on the number of events occurring in the guard (anticoincidence) detectors.

In summary, the information to be received on earth, aside from engineering-type data, is the energy of each scattered alpha particle detected in the alpha mode and the energy of each particle registered in the proton mode of the instrument. The characteristics of these events are to be accumulated on earth and the resulting spectra analyzed in terms of the response of the instrument to pure elements (see below).

NOMINAL MISSION SEQUENCE

In this section will be described a possible experiment sequence, starting with instrument calibration, including pre-flight operations, and finally describing a nominal mission to the moon.

The basic preflight information required for the experiment is a library of spectra, giving the response of a typical instrument to pure elements, in both alpha and proton modes. Such a library will include the response to all elements between boron and titanium (except neon, argon, and scandium) and to selected heavier elements, such as iron, barium, and gold. Typical response curves, obtained with one of the prototype instruments, are shown in Fig. 2 and 3. The instruments are designed to be geometrically identical as regards the paths of scattered alpha particles and protons. Different instruments can be expected to differ slightly, however, in electronic gain, thresholds of response, quality of sources, and thickness of gold absorbers over the proton detectors. These parameters can affect slightly the detailed response of an instrument, and so must be established before a mission in order to make most effective use of the library. Moreover, the temperature of the instrument, when operating on the lunar surface, may vary considerably (estimates range from -40°C to $+50^{\circ}\text{C}$). Thus, the temperature coefficients of the electronics (gain and threshold) and of the detector response have to be determined. The instruments constructed so far have had temperature coefficients of up to 6% over a 90°C temperature range.

During an actual mission, there will be three independent checks on the instrument performance. Firstly, the preflight calibrations will allow an estimate of the energy scale to be made from the telemetered temperature of the unit on the moon.

Secondly, the electronic pulser, upon command from earth, will provide a two-point calibration of each electronic system. Finally, a small amount of purposely placed alpha-radioactive material on each of the detectors will give a one-point calibration of each system. Es^{254} is used for this purpose. Its energy (6.42 MeV) is high enough, and the amounts used are small enough ($\sim 1 \text{ event min}^{-1}$) to preclude interference with the spectra of primary interest.

The behavior of the silicon semiconductor detectors is less reliable than that of other components, and therefore the instrument has been designed with parallel detectors (two alpha and four proton). These can be individually calibrated. In addition, in case of malfunction, the outputs of the separate detectors can be electronically blocked by command from earth.

The long test periods associated with space missions, combined with the relatively short half-life of Cm^{242} (163 days), will make it necessary to install a new set of sources shortly before the start of the mission. This source replacement can be performed from the top of the instrument to minimize contamination of the sensitive part of the equipment.

A possible sequence of operations after landing on the moon might be as follows: The instrument power will be turned on and a calibration sequence performed, using the electronic pulser. At this stage, the instrument will be supported on the spacecraft on a sample of known composition. A measurement lasting approximately three hours will be made on this sample.

At the completion of this phase of operations, the instrument will be deployed onto the lunar surface in two stages. At first, it will be suspended about 15" above the nominal surface, and the response will be noted in this position. At this height, the geometry for alpha particles or protons to get back to the detectors is very small. The data obtained in this position will thus give information on the background rates in the instrument due to cosmic rays or solar protons. The instrument will then be lowered onto the lunar surface and a measurement made for at least 24 hours. Interspersed with this data accumulation will be calibrating sequences, using the electronic pulser.

A companion experiment planned for the Surveyor Scientific Mission is the Soil Mechanics-Surface Sampler Experiment [R. Scott, 1966]. The claw device of this instrument has the capability of picking up the deployed head of the Alpha-Scattering Instrument and placing it down on another area of the lunar surface. The successful accomplishment of this operation would allow a second chemical analysis to be performed on the same mission.

EXAMPLES OF RESULTS OBTAINED WITH INSTRUMENT

The first prototype of this instrument has been used to check the ability of the technique to obtain useful analytical information about complex rocks. Using an early prototype, a library of the response of the instrument to twenty chemical

elements was prepared by measurements on either pure elements or simple compounds. Samples of rocks, in powdered or slab form, were then presented to the instrument in a vacuum at room temperature. Measurements were made for periods of time and with source strengths comparable to those which might apply on a lunar mission.

Figure 4 illustrates the type of data obtained with one of the samples (Hamlet -- a chondritic meteorite). The qualitative features of the alpha spectrum immediately identify the presence of oxygen and the lack of significant amounts of elements with atomic weights much greater than that of iron. A quantitative analysis of the spectra (both alpha and proton) was made, using the library of elemental spectra and least-squares computational techniques, as described by Patterson, Turkevich, and Franzgrote (1965).

Before performing the least-squares analysis, the spectra were corrected for slight instrument drifts. In the cases treated here, this was done from the record of the instrument response to monochromatic alpha sources measured periodically during the course of the measurements. These corrections for changes in instrument gain were less than a few per cent. Likewise, the data were corrected for backgrounds in the instrument. These were much less than ten per cent of the spectra observed, except at the highest energies.

As described in the report of Patterson et al. (1965), the interpretation of complex spectra in terms of a chemical analysis usually involves the assumption that the atomic ionization energy loss of alpha particles in different elements varies as the square root of the mass number. This assumption was made in the work reported here. In addition, in the results described in this report, a library of only thirteen elements was used. At this stage of the work, this was considered to be a reasonable compromise between the desirability of a larger number of components and the consequences of the known, somewhat poorer than desirable, quality of the sources used in these particular measurements. (Later, less extensive work has confirmed the expectation that higher quality sources with the same instrument give spectra with more detailed structure and, therefore, higher information content.)

Table I shows the results obtained by this technique on eight rocks of a reasonable range in composition. The data are presented in terms of atomic per cent, together with a measure of the uncertainty. This latter is derived solely from the statistical errors of the measurement and the least-squares analysis. The table also presents results of analyses obtained by more conventional techniques.

Table II gives a statistical summary, for the most important elements examined, of the degree of agreement between the results obtained with this type of instrument and those from standard methods of analysis. The table lists, for each element, the standard deviation, in atomic per cent, of the two types of analysis. The results for most of the elements indicate absolute accuracies for this method of about one atomic per cent. The good results for sodium and aluminum depend strongly on the data from the proton mode of the instrument and emphasize the need to maintain the capability of this mode of operation. The rather larger deviations for oxygen come primarily from two samples (the tektite and the meteorite) and may represent an inadequate calculational treatment of the data.

Although these results cover a very small number of rocks, they clearly establish the capability of the instrument to distinguish between gross classes of terrestrially occurring materials. In addition, the presence of unusual amounts of elements, such as nitrogen or fluorine, should easily be seen. Finally, the high sensitivity of this technique for carbon should ensure the detection of amounts of this element comparable to those found in carbonaceous chondrites.

The long times of operation needed for a complete analysis by this technique has led to a study of the types of information that could be extracted from a limited amount of data (resulting, for example, from power failure or overheating on the lunar surface). Figure 5 illustrates, for the alpha mode, the statistical

nature of the data obtained in 20 minutes and 243 minutes, as compared to a complete 2400 min. observation on a sample of a basalt. It is clear that even the short period of observation would identify the presence of oxygen and the general nature of the electropositive element (sodium, magnesium, aluminum, or silicon rather than iron). The quality of the results improves continuously with length of operation.

These results thus confirm the expectation that this type of instrument, if placed on the moon, will provide scientifically significant information concerning the chemical nature of the lunar surface.

ACKNOWLEDGMENTS

The planning, instrumentation, and implementation of this experiment have involved contributions by many different people. At the University of Chicago, the various technical groups of the Laboratory for Astrophysics and Space Research, led by James E. Lamport, Wayne A. Anderson, and A. J. Tuzzolino, have been indispensable, as have been the calculations performed by Ken Sowinski. Dale E. Suddeth, of the Argonne National Laboratory, has contributed to the electronics developments, and Glenn Sisk and Tim Harrington, of the Jet Propulsion Laboratory, to the planning of this experiment.

The work at the University of Chicago was supported in part by grant NSG-127-61 of the National Aeronautics and Space Administration and by subcontracts JPL-NASA #950315 and #950750 with the Jet Propulsion Laboratory. The work of the Jet Propulsion Laboratory was supported by contract NAS 7-100 of the National Aeronautics and Space Administration. The work at Argonne National Laboratory was performed under the auspices of the United States Atomic Energy Commission.

REFERENCES

- Patterson, J. H., Turkevich, A. L., and Franzgrote, E., Chemical analysis of surfaces using alpha particles, J. Geophys. Res., 70, 1311, 1965.
- Scott, R., The soil mechanics-surface sampler experiment for Surveyor, (in preparation), 1966.
- Turkevich, A. L., Chemical analysis of surfaces by use of large-angle scattering of heavy charged particles, Science, 134, 672, 1961.
- Turkevich, A. L., Knolle, K., Emmert, R. E., Anderson, W. A., Patterson, J. H., and Franzgrote, E., An instrument for lunar surface chemical analysis. Submitted to The Review of Scientific Instruments, May, 1966.
- Walker, G., Wolf, L., and Kostenko, C., Thermal aspects of the alpha scattering device, I.I.T. Research Institute Report, September, 1965.

FIGURE CAPTIONS

- Fig. 1. Type Approval Units of the Alpha-Scattering Instrument (Prototype #3). The head unit is on top — with the 12" plate to provide a larger area-bearing surface; the electronics unit is on the bottom.
- Fig. 2. Examples of Elemental Response Curves of Instrument in Alpha Mode. The ordinates give the events in each channel in a 1000 min. measurement, with a total source strength of 6.7×10^{10} d/m of Cm^{242} . The ordinates are channel numbers of the pulse-height analyzer (linearly related to the energies of the backscattered alpha particles). The data for oxygen were obtained from those for oxides by the methods of Patterson, Turkevich, and Franzgrote (1965).
- Fig. 3. Examples of Elemental Response Curves of Instrument in Proton Mode. The data were obtained with a source strength of 6.7×10^{10} d/m. The data for sodium were obtained from measurements on Na_2CO_3 by the methods of Patterson, Turkevich, and Franzgrote (1965).
- Fig. 4. Response of Instrument to Sample of Chondritic Meteorite (Hamlet). The ordinates for the Alpha and Proton modes are on the right and left, respectively. The data apply to a 1000 min. measurement time, with a source strength of $\sim 7 \times 10^{10}$ d/min.

Fig. 5. Effect of Measurement Duration on Quality of Results.

The data are for the response in the Alpha Mode to a sample of Basalt. At the top of the figure are indicated the end points of spectra for some pure elements. These data were obtained with sources of higher quality than the data of Figs. 2, 3, and 4.

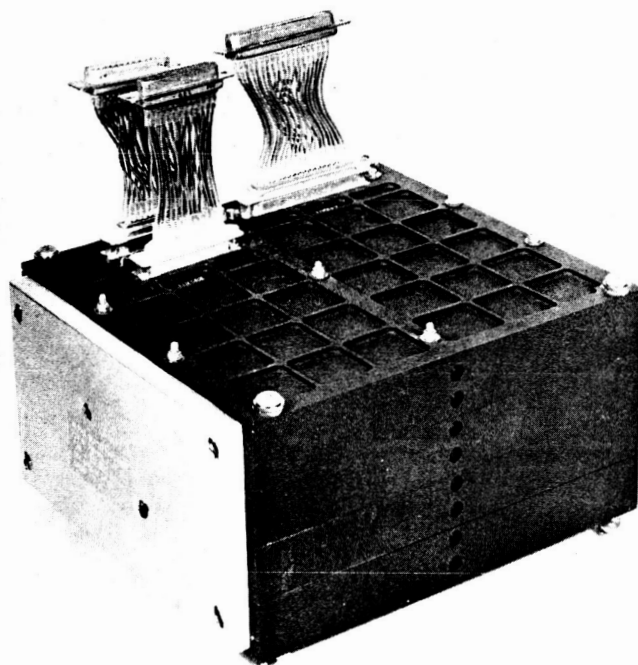
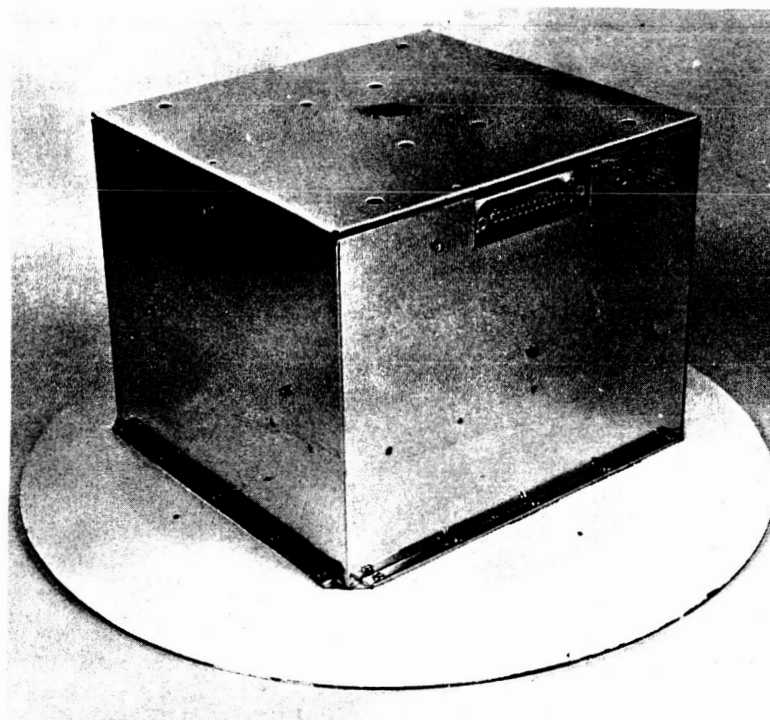


Fig. 1

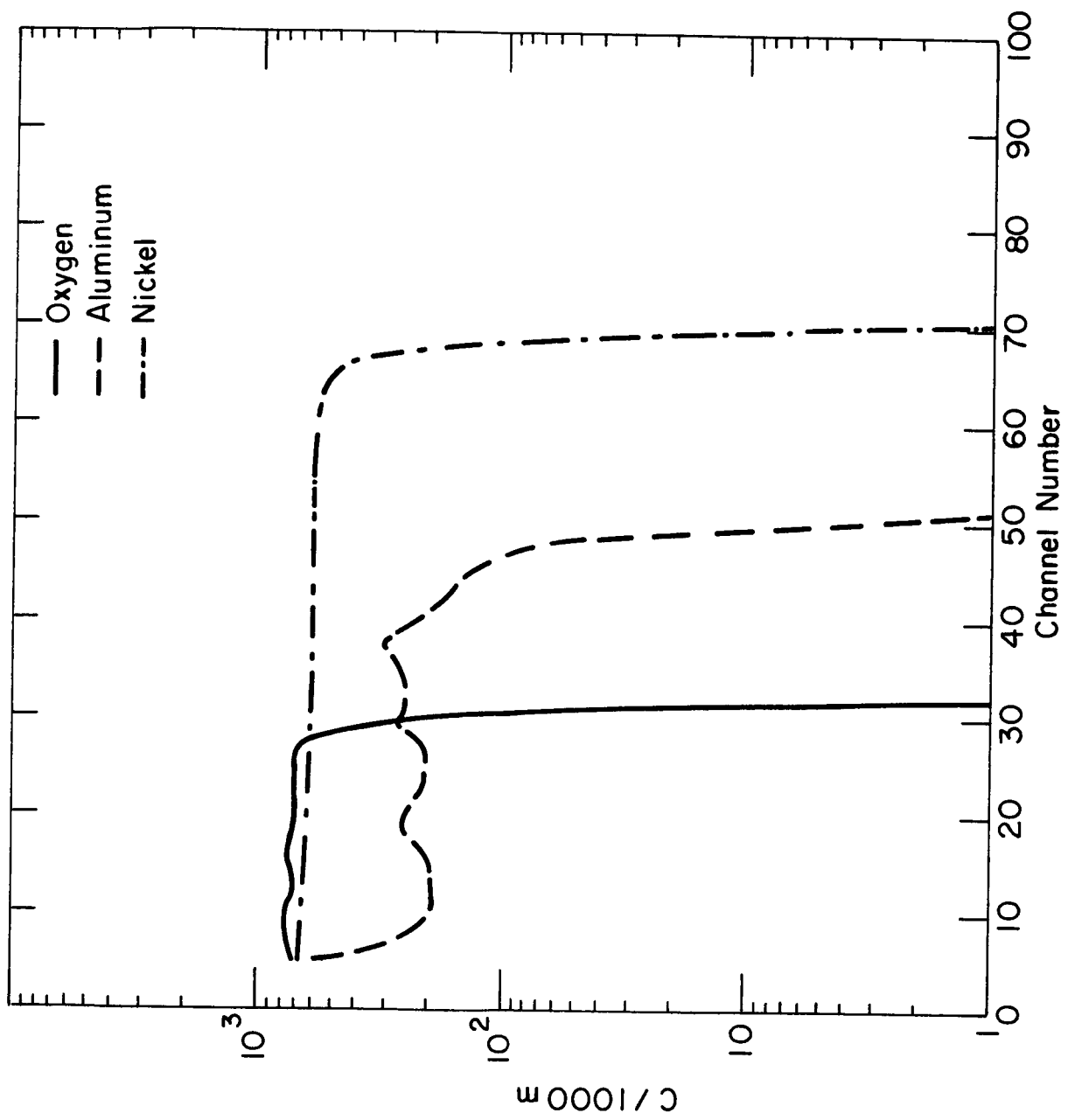


Fig. 2

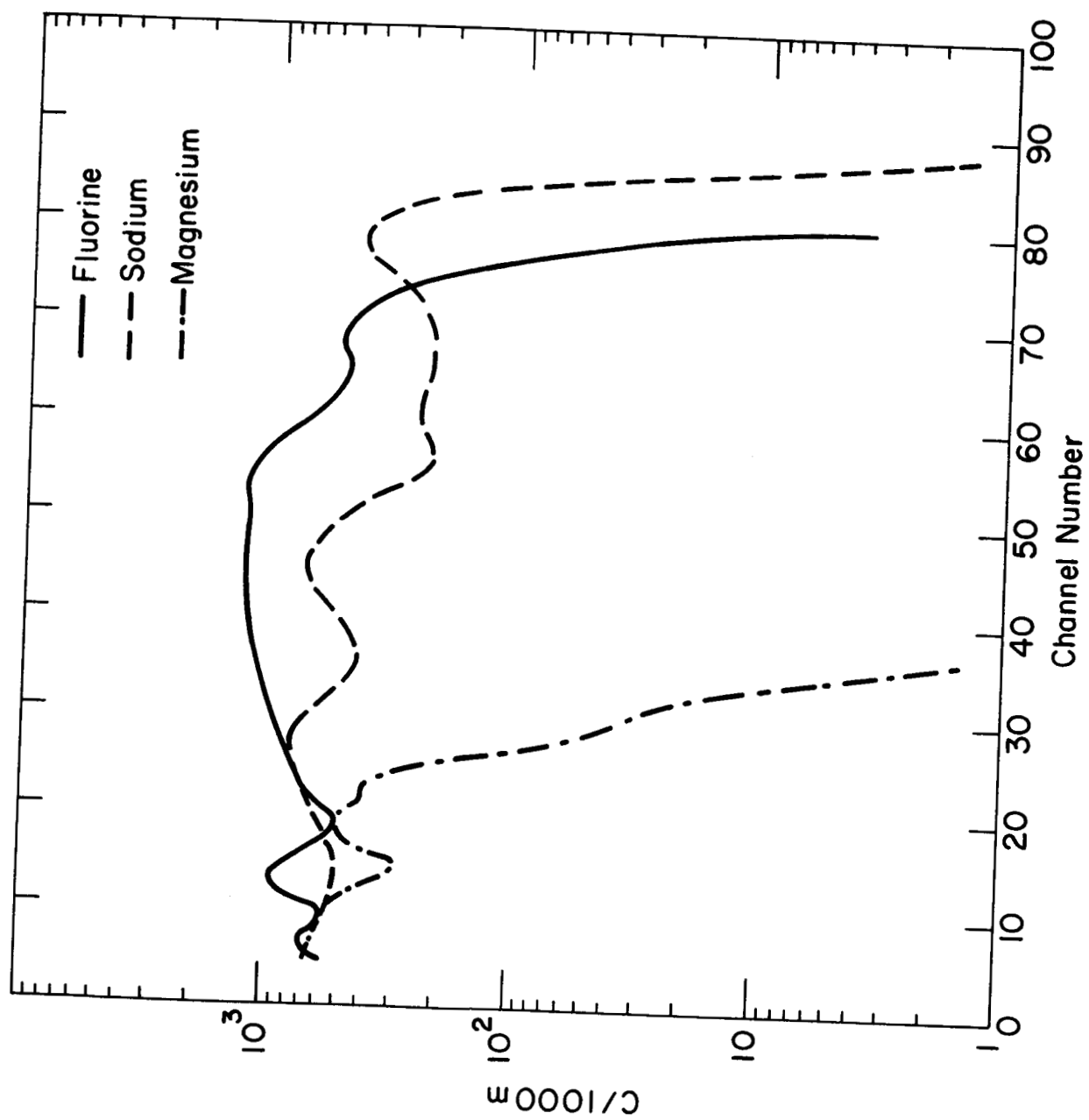


Fig 3

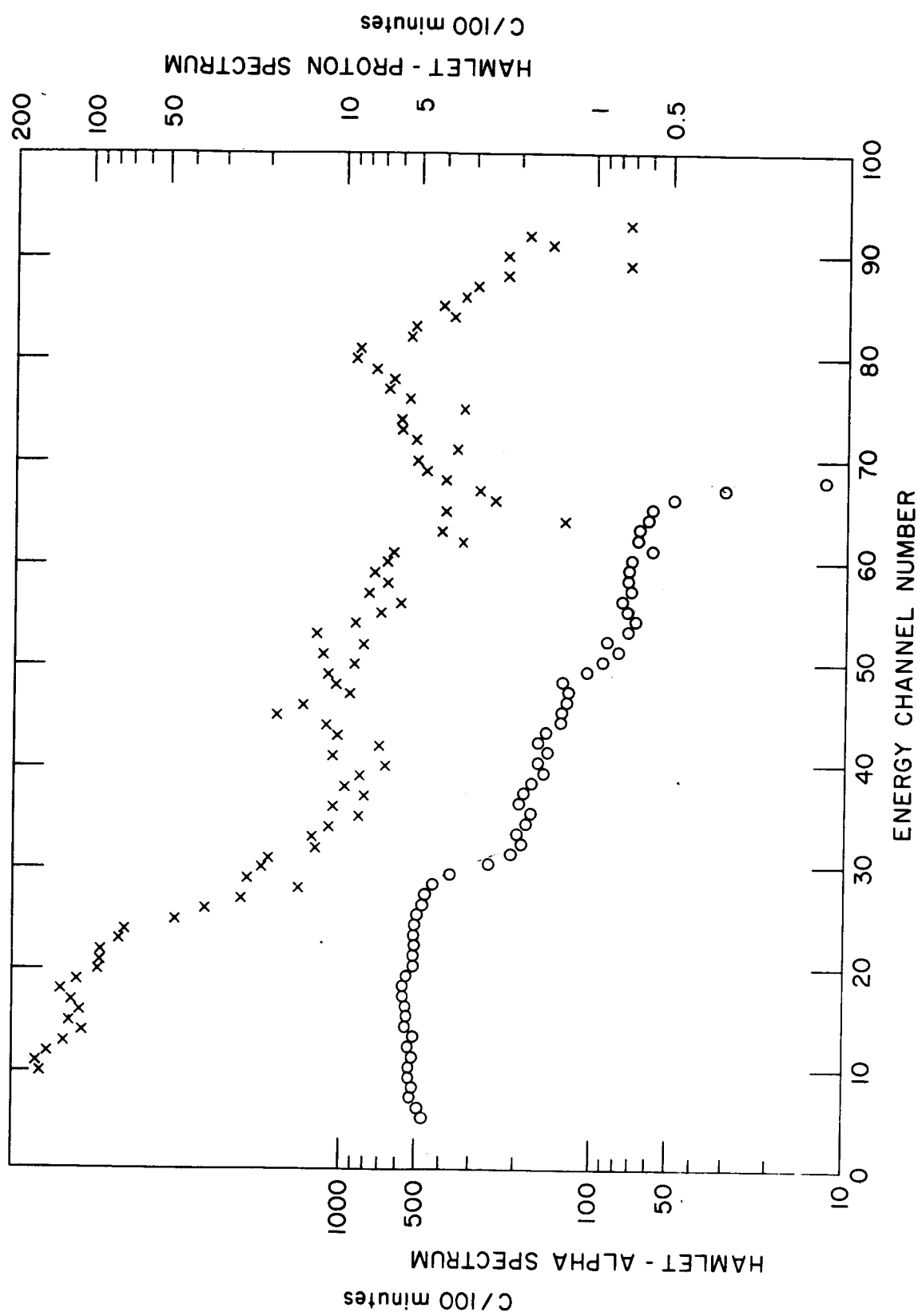


Fig. 4

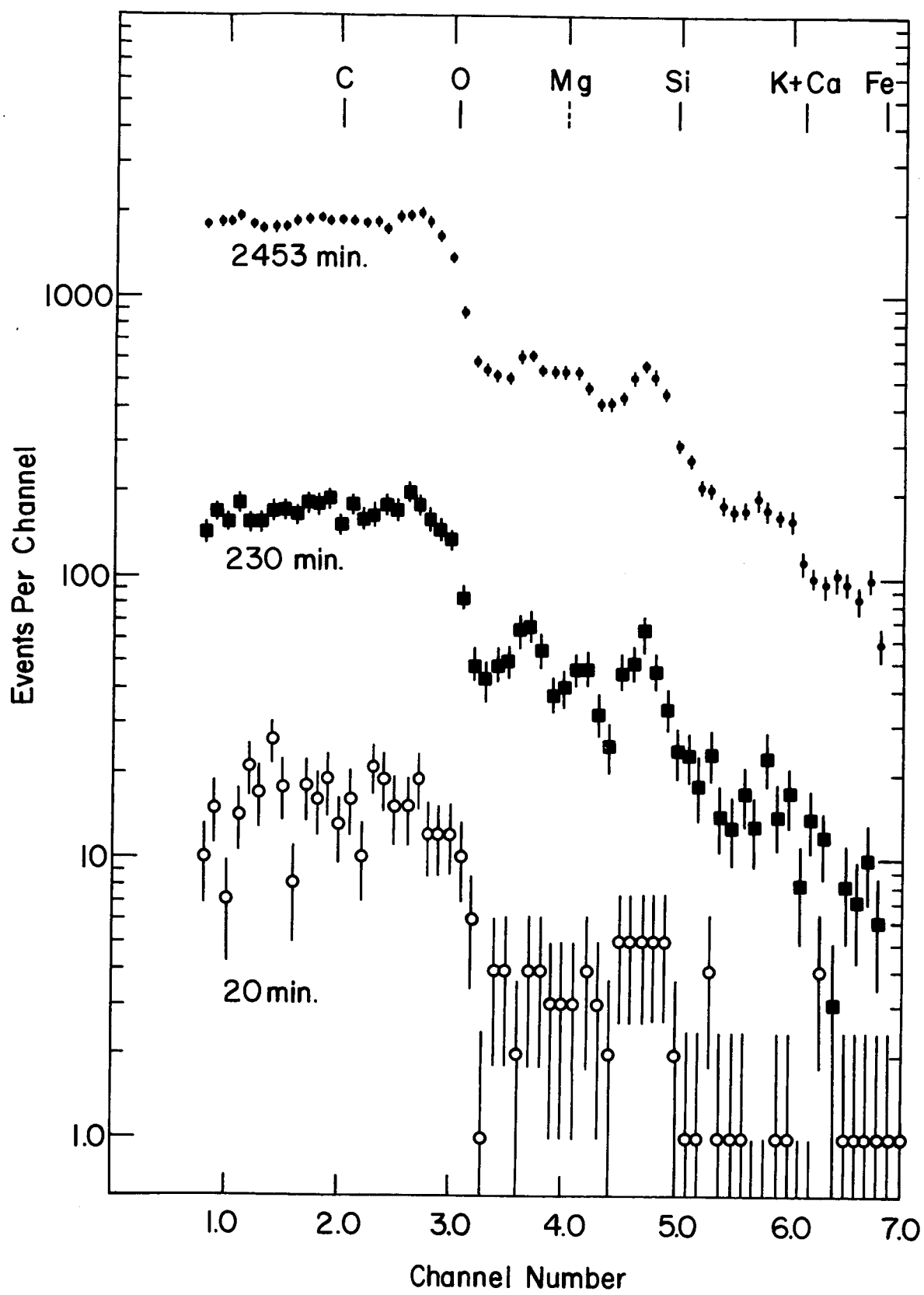


Fig.5

TABLE I. Comparison between Alpha and Conventional Analyses of Some Rocks
(Atomic Fractions)

Rock	W-1			G-1			Dunite			Hamlet		
	Alpha	Conv. ^c		Alpha	Conv. ^c		Alpha	Conv. ^c		Alpha	Conv. ^c	
Hydrogen	-	0.011		-	0.009		-	0.043		-	0.001	
Carbon	0.004 ±0.004	0.0003		0.010 ±0.006	0.0004		a	0.0005		0.006 ±0.003	b	
Oxygen	0.588 ±0.009	0.604		0.618 ±0.013	0.617		0.582 ±0.005	0.560		0.541 ±0.007	0.566	
Fluorine	0.0000±0.0005	0.0002		a	0.0008		0	0.0001		a	b	
Sodium	0.021 ±0.003	0.0144		0.022 ±0.005	0.022		0.0002±0.0006	0.0001		0.0075±0.0014	0.0078	
Magnesium	0.018 ±0.008	0.0354		0.011 ±0.012	0.002		0.194 ±0.005	0.231		0.128 ±0.006	0.148	
Aluminum	0.066 ±0.004	0.0628		0.059 ±0.006	0.057		0.0012±0.0007	0.0019		0.013 ±0.002	0.0123	
Silicon	0.203 ±0.009	0.189		0.235 ±0.012	0.256		0.150 ±0.006	0.139		0.159 ±0.007	0.162	
Phosphorous	a	0.0004		a	0.0003		0.0002±0.0006	0		a	0.0014	
Sulfur	0.020 ±0.009	0.0001		0.010 ±0.012	0.0001		0.023 ±0.005	b		0.038 ±0.007	0.0145	
Chlorine	a	b		0.016 ±0.008	0.0001		a	0.0001		0.001 ±0.004	b	
"Calcium"*	0.034 ±0.006	0.045		0.018 ±0.006	0.029		a	0.001		a	0.0092	
"Iron"*	0.031 ±0.002	0.0378		0.003 ±0.001	0.0059		0.0238±0.008	0.0232		0.084 ±0.002	0.079	
"Barium"*	a	0.0001		0.0003±0.0001	0.0006		a	b		0.0007±0.0004	b	
Rock	Tektite			Limestone ^d			Sulfide Ore ^d			Syenite ^d		
	Alpha	Conv. ^c		Alpha	Conv. ^c		Alpha	Conv. ^c		Alpha	Conv. ^c	
Hydrogen	-	0.008		-	0.164		-	0.0852		-	0.0015	
Carbon	a	0.00006		0.168 ±0.004	0.164		0.007 ±0.003	b		0.003 ±0.004	0.0043	
Oxygen	0.597 ±0.005	0.639		0.593 ±0.008	0.599		0.458 ±0.008	0.461		0.606 ±0.009	0.600	
Fluorine	a	0.0001		a	b		0.0001±0.0004	b		a	b	
Sodium	0.010 ±0.002	0.0073		0.0039±0.0015	0.00255		0.0076±0.022	0.00745		0.024 ±0.004	0.0205	
Magnesium	0.0114±0.0065	0.0144		0.002 ±0.005	0.0111		0.031 ±0.007	0.0259		a	0.0212	
Aluminum	0.042 ±0.003	0.048		0.016 ±0.002	0.0164		0.039 ±0.003	0.0450		0.035 ±0.005	0.0388	
Silicon	0.261 ±0.007	0.252		0.057 ±0.006	0.0473		0.132 ±0.009	0.147		0.225 ±0.012	0.210	
Phosphorous	0.001 ±0.002	0.0001		0.001 ±0.001	0.0005		0.001 ±0.002	0.00033		a	0.0006	
Sulfur	0.006 ±0.007	b		0.013 ±0.006	0.0017		0.056 ±0.009	0.0863		a	0.00047	
Chlorine	0.004 ±0.005	0.00006		0.004 ±0.004	b		a	b		a	b	
"Calcium"*	0.017 ±0.004	0.021		0.154 ±0.005	0.152		0.045 ±0.007	0.0212		0.046 ±0.014	0.0512	
"Iron"*	0.016 ±0.001	0.0163		0.0075±0.0009	0.0047		0.103 ±0.003	0.120		0.031 ±0.006	0.0495	
"Barium"*	0.0003±0.00006	0.00001		a	b		0.00009±0.00006	0.0003		0.0016±0.0007	0.0017	

* See following page for explanation of Footnotes.

TABLE I. Comparison between Alpha and
Conventional Analyses of Some Rocks
(Atomic Fractions)

FOOTNOTES:

*In this table, "calcium" means potassium and calcium;
"iron" denotes the elements titanium through zinc, inclusive;
"barium" means all the elements heavier than zinc.

^aIn these cases, the least-squares calculation gave
slightly negative answers. The value was set to zero and the
calculation repeated.

^bNo results of conventional analyses are available in
these cases.

^cThese results of conventional analyses are from the
U. S. Geological Survey.

^dThese samples (with results of conventional analyses)
were obtained from the National Bureau of Standards.

^eThis analysis of the meteorite Hamlet was performed by
H. B. Wiik.

TABLE II. Statistical Summary of
Comparison between Alpha and Conventional Analysis

(Standard deviation, in atomic per cent,
between the results of the two types of analysis)

Element	Standard Deviation
Oxygen	2.1
Sodium	0.3
Magnesium	2.0
Aluminum	0.4
Silicon	1.3
"Calcium"	1.2
"Iron"	0.7